Effect of thermal shock conditions on reliability of chip ceramic capacitors

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Abstract

Different size X7R MLCCs have been subjected to three types of thermal shock testing: terminal solder pot dip test, ice water test, and liquid nitrogen drop test. Electrical characteristics of the parts were measured through various test conditions to determine critical temperatures that result in fracturing and electrical failures. Optical examinations and cross-sectional analysis were used to confirm the presence of cracks. Mechanisms of fracturing and the effectiveness of different thermal shock methods are discussed. It is shown that the heat conduction and direction of temperature changes (heating or cooling) are critical factors of thermal shock testing of MLCCs. The probability of fracturing depends also on the level of residual mechanical stresses, mostly in terminal areas of ceramic capacitors.

Key words: ceramic capacitors, thermal shock, failure

Introduction

Soldering related thermal shock (TS) is one of the major causes of fracturing in MLCCs that might result in latent defects and cause failures with time during application. The probability of cracking generally increases with the size of capacitors, and is especially high for manual soldering (see literature review in [1]).

Various thermal shock techniques to assess the susceptibility of ceramics to fracture are described in military and industry specifications and in technical literature. The most popular techniques are water quenching, that is typically used for ceramic materials, and solder dip test that is widely used for high-reliability MLCCs and is described in relevant military specifications. Immersion in liquid nitrogen is often used to assess thermo-mechanical reliability of the parts intended for cryogenic applications.

Solder dip test provides thermal shock conditions that closely simulate stresses related to manual soldering. However, clamping of the parts or mounting in a fixture for plunging into molten solder might have a strong effect on test results [2]. GEIA-STD-0006 (2008) that was developed with the purpose of setting industry-wide requirements for solder dip to replace finishing on electronic parts emphasizes that the controls needed for the dip process are not well enough understood to be included in an industry standard [3].

The water quenching technique has been used for ceramic capacitors by several authors. Koripella [4] studied fracture toughness, modulus of rupture, and thermal shock resistance on COG, X7R, and Z5U type materials. The 1206 size COG and X7R samples showed a sudden drop in the strength at a temperature difference of 150 °C and Z5U samples at a temperature difference of 125 °C. These values were close to the predicted critical temperatures (thermal shock resistance parameter).

Chang and co-authors [5-7] used ice water test (IWT) to study fracturing in multilayer ceramic capacitors of different size. It was shown that the thermal shock resistance decreases in a row 0402, 0603, 0805, and 1206 capacitors, and the corresponding critical temperatures are 400 ºC, 300 ºC, 200 ºC, and 100 ºC. TS resistance of Y5V and Z5U 0.1 uF 0805 size capacitors was shown to be inferior compared to similar size and value of BT parts. Yeung et al. [8] confirmed that smaller capacitors have higher TS resistance than the larger ones. However, the dependence of thermal shock resistance on the thickness differ substantially from the one predicted by calculations, likely due to the presence of preexisting flaws.

Considering a wide use of manual soldering for space applications, there is a need for better understanding of the effect of TS on MLCCs and in development of adequate test methods that would assure the robustness of MLCCs at soldering conditions. The purpose of this work is to get more insight into the mechanism of the effect of TS on MLCCs and to compare the effectiveness of different thermal shock methods. For this purpose various types of large-size MLCCs were subjected to the terminal solder pot testing, ice water testing, and liquid nitrogen drop testing. Measurements of electrical characteristics of the parts, optical examinations, and cross-sectioning were used to assess the effect of different TS conditions.

Experiment

Sixteen types of multilayer ceramic chip capacitors from five vendors were used in this study (see Table 1). All parts employed X7R dielectric materials, nine parts were rated to 1 μF 50 V, five to 0.1 μF 100 V, and two to 10 μF 50V.

Table 1. Capacitors used for thermal shock testing

Gr	MLCC, uF/V	Mfr	Size	L (mm)	W (mm)	H (mm)
1	1/50	A	1210	3.29	2.61	2.02
2	1/50	A	1812	4.38	3.13	1.14
3	1/50	A	2220	5.76	5.36	1.72
4	1/50	B	1210	3.1	2.51	1.54
5	1/50	B	1812	4.3	3.1	1.28
6	1/50	B	2225	5.57	6.24	0.92
7	1/50	C	1206	3.29	1.74	1.66
8	1/50	C	0805	2.02	1.27	1.28
9	1/50	C	2220	5.78	4.82	1.89
10	10/50	A	2220	5.78	4.94	2.52
11	10/50	T	2220	5.87	5.34	2.26
12	0.1/100	A	1825	4.38	6.31	1.11
13	0.1/100	A	1812	4.43	3.11	0.92
14	0.1/100	P	1825	4.65	6.53	1.47
15	0.1/100	A	1210	3.2	2.5	1.5
16	0.1/100	A	1210	3.2	2.5	1.5

The parts were subjected to terminal solder dip test (TSD) at solder pot temperatures varying from 300 °C to 350 °C; ice-water quenching test at preheat temperatures from 150 °C to 250 °C, and liquid nitrogen (-197 °C) drop test. Details of the techniques used are described in the sections below. Vicinal optical illumination microscopy [9], measurements of capacitance, dissipation factor and leakage currents were used before and after each test.

Terminal solder dip test

Four types of large-size capacitors from Gr.3, Gr.6, Gr.12, and Gr.14 with ten samples each were used for this test. One terminal of each capacitor was dipped for 5 seconds into a pot of molten solder heated initially to 300°C and then the part left to cool at room temperature for 5 minutes. This solder dip and cooling procedures were performed 10 times for each part, after which all capacitors were tested for leakage currents. To enhance moisture sorption in possible cracks, post TS measurements were repeated after allowing samples to store at room conditions for one week. Then the solder dip test was repeated at molten solder temperatures of 325°C and 350°C.

Terminal solder pot test that have been used in this study provides more adequate test conditions compared to conventional solder dip testing. During this test one terminal of the capacitor is clamped in a fixture while the other is touching molten solder. This eliminates local pressure to ceramic and unpredictable changes in temperature distribution caused by application of tweezers, stabilizes temperature at the clamped terminal, and creates a significant temperature gradient at another terminal.

Figure 1 shows results of the testing for Gr. 3 and Gr. 6 of capacitors. No significant difference in leakage currents was observed before and after the solder shock test at 300°C and the currents remained stable after one week of storage. Ten additional solder dip cycles after solder temperature was increased to 325°C also did not result in degradation. For three part types, Gr.6, Gr.12, and Gr.14 no change in currents was observed even after 10 solder dip cycles at 350 °C. Microscopic examinations did not reveal any cracks in capacitors from Gr.6 and Gr.12. Some variations of capacitance and dissipation factors were consistent with the de-ageing process due to the temperature of the parts exceeding the Curie point.

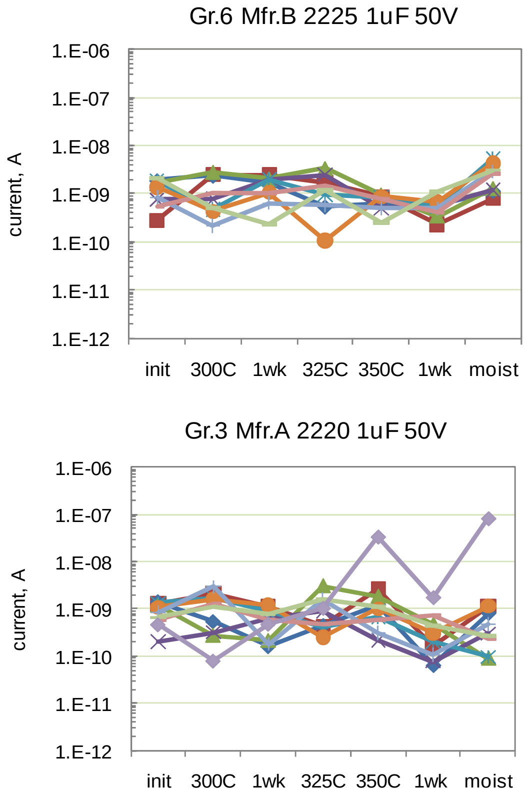


Figure 1. Variations of leakage currents during TSD testing in two groups of capacitors.

After 350 °C solder dip test one out of ten samples in Gr.3 increased DCL almost by two orders of magnitude (see Figure 1b), whereas all other parts in this group remained stable and did not degrade even after storage in humid environments (3 days at room temperature and 98% RH). Microscopic examinations confirmed the presence of cracks near terminals in the failed sample.

Results of this study show that large-size capacitors (2220 and 1825) have high resistance against thermal stresses developed during solder dip testing. No failures were observed in 40 parts after 10 cycles at 300 °C and 325 °C, and only one failure occurred after testing after 350 °C cycling. This is in agreement with the results of our previous study [1] that showed no failures in 70 samples from seven lots of large, 2220 size, parts tested by 100 terminal solder dip cycles at a solder temperature of 300 °C.

Ice water test.

Ten samples from each of the 16 groups shown in Table 1 were placed on a hot plate, covered with a Teflon cover, heated to a desired temperature and stabilized for 10 minutes. After stabilization, capacitors were rapidly quenched into water by sweeping the cover and dropping the parts from a hot plate into a nearby bath with ice water. Leakage currents were measured within one hour after IWT. The temperature of the hot plate was set to 150°C initially and then increased by 25°C increments for consecutive test cycles.

Examples of variations of leakage currents through IWT for three types of capacitors are shown in Figure 2 and clearly indicate the presence of a critical temperature when DCL increases substantially, two to five orders of magnitude. Most of the parts in 14 groups, except two parts in Gr.13, could withstand IWT at 150 °C without any significant changes in leakage currents, but none of the parts withstood TS at more than 225 °C.

To evaluate for how long this high-leakage-current state remains, the parts were stored at room conditions, and leakage currents were measured after one day, one week, two weeks, and one month of storage. In most cases a significant decrease in DCL was observed already after one day, but for some parts the currents gradually decreased during several weeks. In six groups (Gr. 1, 3, 4, 11, 12, 13) from 10% to 70% of the samples remained at a relatively low insulation resistance (IR) state even after one month of storage. The highest level of leakage currents after 1 month was in Gr.3, G.12, and Gr.13 that all had silver/palladium inner electrodes. All lots except for Gr.7, Gr.8, and Gr.14 had DCL values at least an order of magnitude greater than initially even after a month of storage.

Median values of IR after crack formation and water exposure are typically in the megohm range. Minimal values are in the hundredths of kiloohm to megohm range, and additional measurements showed that none of the parts failed at IR below one kiloohm.

Approximately 5% to 10% increase in capacitance that was observed after the first test and decrease after one week of storage are due to de-aging/re-aging processes in barium titanate ceramics. However, no substantial variation in C and/or DF occurred in most of the parts even when the preheat temperature reached the critical level resulting in high DCL. These confirms that AC characteristics are much less sensitive to TS testing compared to leakage currents, which is in agreement with results of Chan and co-workers [10].

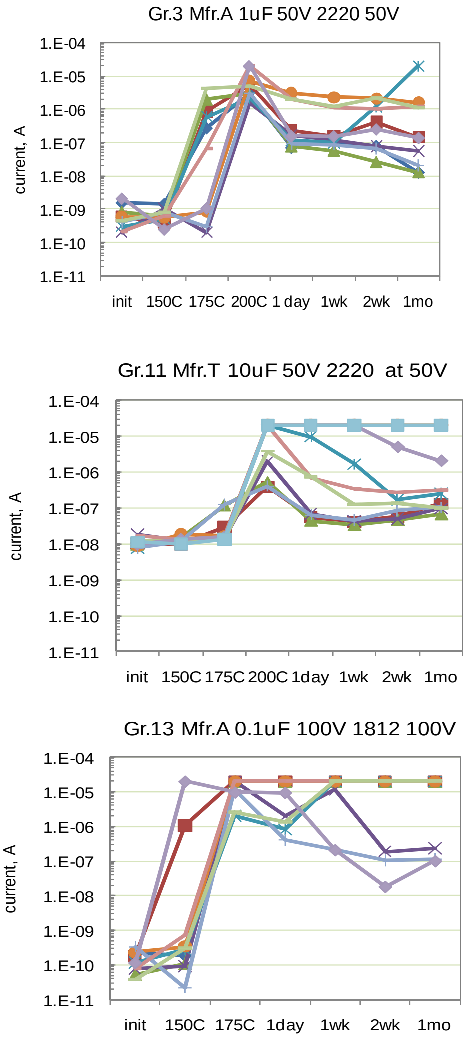


Figure 2. Variations of leakage currents through the ice-water-testing at different temperatures and storage conditions for three lots of MLCCs

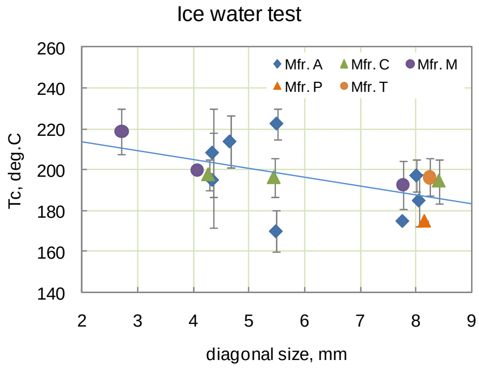
Average values of the critical temperature, ΔT_{c}, and standard deviations determined based on results of IWT for ten parts are shown in Table 2. The minimal value of ΔT_c = 170 °C was observed for Gr.13 parts, and maximum, 222.5 °C, for Gr.2. The standard deviations in all cases were relatively small varying from 0 to 22.3 (average value is 9.6 °C) thus indicating a relatively small spread and good reproducibility of test results. Repeat testing of 19 samples from Gr.1 capacitors resulted in average ΔT_c = 209.2 °C and SDT = 18.5, which is close to the data presented in Table 2 and indicate good reproducibility of the IWT. Decreasing temperature increments during the testing and increasing sample size would provide more distinctive results and allow for better assessment of the critical temperature.

Values of capacitors, the type of metal electrode used and/or type of termination are apparently not critical factors for ice water thermal shock testing.

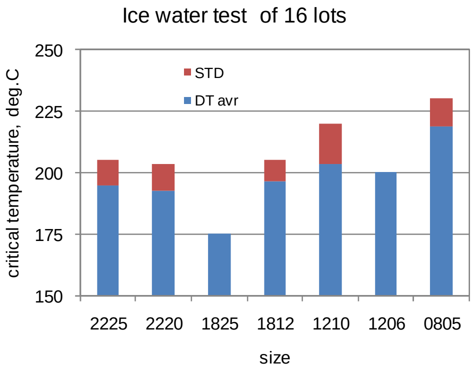
Table 2. Critical temperatures (in deg.C) per IWT.

Gr.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
T_crit. avr.	210	222.5	185	197.5	196.1	194.4	200	218.8	192.5	197.2	196.3	175	170	175	208.3	195
STD	11.5	7.8	12.6	7.8	9.4	10.7	0	11.1	11.8	8.1	9.2	0	10.3	0	20.9	22.3

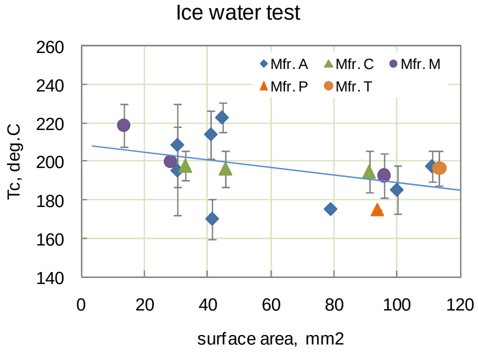
Analysis shows that none of the geometrical factors (size, thickness, and surface area of the parts) have a strong correlation with the critical temperature. In particular, no correlation between the thickness of the capacitors and ΔT_c as it is predicted by a simple TS model was observed. Figure 3a shows a trend of decreasing of the thermal shock resistance with the overall size of parts.



(a)



(b)



(c)

Figure 3. Correlation between the critical temperature of IWT and standard size (a), diagonal size (b), and the surface area of the parts (c).

However, the correlation with the standard size (see Figure 3b) is not as good as reported in [7]. A somewhat better correlation exists between ΔT_c and the surface area of the parts, which is in agreement with the results presented by Yeung at al. [8] and likely indicates the presence of surface flaws in the capacitors.

Optical examinations showed that the cracks originated mostly at the corners/edges of the capacitors, often at the termination edge and there was a trend of increasing the number of cracks with the surface area. This is in agreement with the correlation between ΔT_c and the surface area and with a trend of decreasing values of post-IWT IR for large ceramic capacitors.

Results of cross-sectioning confirmed the presence of cracks crossing inner electrodes of the parts. In most cases the cracks were located at the terminal areas (see Figure 4).

Figure 4. Cross-sectioning views of samples after IWT at critical conditions showing locations of cracks.

Liquid nitrogen drop test.

Four types of multilayer ceramic chip capacitors from Gr.1, Gr.3, Gr.6, and Gr.7, with 10 samples each were used in this experiment. Capacitors that were thermally stabilized at room temperature were dropped into a Dewar with liquid nitrogen (LN) and then removed into a sealed plastic bag to avoid moisture condensation and icing. After stabilizing at room temperature the parts were removed from the bag for electrical characterization. Because the difference between LN (-197 ^{o}C) and room temperature is ~220 ^{o}C, based on results of IWT, it was expected that the LN drop test is sufficient to cause fracturing in majority of the capacitors. However, results (see an example in Figure 5) showed no significant variations of DCL and some decrease in capacitance (~2%) that was likely due to the effect of mechanical stresses [11] associated with exposure to extremely low temperature.

One of the reasons that LN drop test did not increase leakage currents, might be related to the lack of exposure to moisture. In the absence of moisture DCL does not increase substantially even in fractured capacitors. To reveal possible cracks, the samples were stored in a humidity chamber at 85°C and 85% relative humidity for 10 days. However, post-moisture-exposure measurements showed no notable changes in leakage currents and restored initial values of capacitance.

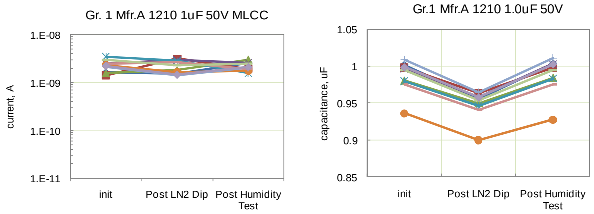


Figure 5. Variations of leakage current and capacitance in Gr.1 ceramic capacitors after liquid nitrogen drop test and exposure to 85 ^{o}C/85% RH for 10 days.

Vicinal illumination microscopy did not reveal cracks in capacitors from Gr.1. One tiny crack was found in one out of 10 parts in Gr.3. Two parts from Gr.7 had one small crack each. However, 9 out of ten parts had one or more cracks in Gr.6. Considering that Gr.6 capacitors have rather large margins (~ 300 μm), a relatively shallow cracks that formed in these parts would not cause any degradation of leakage currents even after moisture exposure.

The results show that thermal shock test caused by quenching in liquid nitrogen resulted in much less severe damage in ceramic capacitors compared to ice-water testing performed at conditions with similar temperature variations.

Discussion

Terminal solder dip testing did not produce failures up to 325 ^{o}C in four lots of capacitors tested in this study. This is consistent with our previous results [1] and with the work by Chan et al. [6] where no failures were observed in parts stressed by solder dip testing at 275 ^{o}C and some decrease in breakdown voltage appeared after testing at 425 ^{o}C only.

During solder dip, a ceramic capacitor experiences a hot thermal shock with an instant temperature increase that results in large transient compressive stresses at the surface and relatively small tensile stresses in the bulk of the specimen. Although this test provides good heat transfer condition and results in formation of significant thermo-mechanical stresses, the probability of fracture and crack formation is low. This is mostly due to a high compressive strength of ceramic materials that is typically in the range from 1 GPa to 4 GPa and is approximately an order of magnitude greater that the tensile strength [12].

Another factor that likely mitigates the effect of solder dip stress and makes MLCCs generally robust enough to soldering induced TS stresses is increased thermal conductivity of capacitors along the metal plates. Our estimations showed that the for BME capacitors with the thickness of the dielectric layer of 10 μm, the effective thermal conductivity increases 2 to 6 times. For PME parts this increase is larger, from 9 to 20 times, and substantially reduces temperature gradients and stress transients in the parts.

An obvious explanation for the increase of DCL during IWT at a critical temperature is that it is caused by fracturing of the parts and formation of a conduction path by moisture in cracks crossing internal electrodes of the capacitor. Contrary to solder dip testing, IWT creates tensile stresses at the surface area of the parts and considering that ceramic materials have a relatively low strength under tensile conditions, this enhances fracturing during cold TS testing compared to hot TS test.

Analysis of mechanical characteristics of barium titanate ceramics and X7R type MLCCs reported in literature [4, 6, 13-14] shows that average values of the tensile strength, σ_c, are in the range from 70 MPa to 250 MPa, a typical values of the Young's modulus, E ~ 100 GPa, coefficient of thermal expansion, α ~ 1e-5 1/K, and Poisson's coefficient, ν = 0.33.

The critical temperature during water-quenching test can be described by the relation [15]:

$$\Delta T_c = \frac{\sigma_c(1-\nu)}{\alpha \times E} \times \left[1.5 + \frac{3.25}{Bi} - 0.5 \times \exp\left(-\frac{16}{Bi}\right)\right],$$

where Bi is the Biot number that characterizes transient heat transfer processes in solids,

$$Bi \equiv \frac{h \times H}{\lambda},$$

λ is the thermal conductivity of ceramic, H is the half thickness of the capacitor, and h the coefficient of heat transfer.

A possible range of heat transfer coefficients, h, is likely in the range from 1,000 W/m^2_K to 30,000 W/m^2_K [16]. At the thickness of ceramic capacitors from 0.06 mm to 4 mm these values of h correspond to Biot numbers from 0.01 to 22.

Using typical mechanical characteristics for X7R ceramic, variations of the critical temperature with Bi for are shown in Figure 6. For the expected range of Bi during IWT, from 3 to 10 [16], ΔTc varies from 78 ^{o}C to 390 ^{o}C which overlaps the experimentally observed values from 170 ^{o}C to 222 ^{o}C. Estimations showed that the expected tensile strength of different X7R MLCCs used in this study varies in the range from 110 MPa to 200 MPa, which is consistent with literature data.

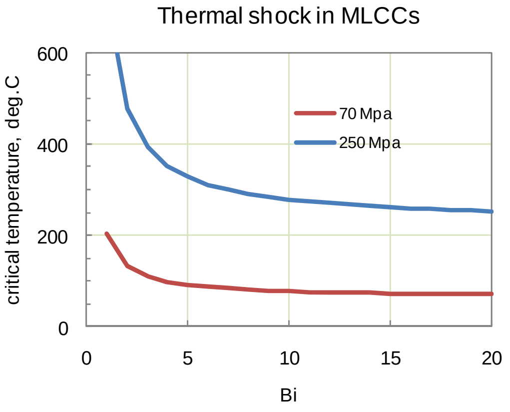


Figure 6. Estimated range of ΔTc for ice-water testing of MLCCs.

One of the factors that complicate analysis of thermal shock conditions on fracturing in MLCCS is the presence of residual or built-in mechanical stresses that are especially large at the edge, termination areas of the parts. These stresses are responsible not only for structural failures such as cracks, delaminations, and deformation, but also for modification of the dielectric behavior of ceramic materials [11]. Because the CTE of barium titanate ceramics is less than that of nickel, during the cooling from sintering (~1300 ^{o}C) to room temperature, the nickel electrode contracts more significantly than the barium titanate layer. This results in tensile stresses in the nickel layer and compressive stresses in the barium titanate layer. The latter might play a positive role by blocking crack propagation caused by cold thermal shock conditions [17]. Stresses in the terminal areas are attributed to different contraction of the terminal region compared to the active layer region in the MLCC. A significant variation of residual stresses from~ -200 MPa (compressive) in the middle area to ~+15 MPa (tensile) at the edge, terminal areas of capacitors, has been demonstrated by finite element analysis and confirmed experimentally by Toonder at al. [18].

Figure 7 shows a trend of decreasing of ΔTc with the periphery of capacitors. This is consistent with the location of increased tensile stresses and with the fact that the majority of cracks are generated at the terminal areas. This indicates also that residual mechanical stresses are playing an important role in fracturing of MLCCs under thermal shock stress.

Although cold TS conditions during IWT do not simulate directly thermal shock stresses specific for initial phases of soldering, it allows assessing the probability of fracturing during the cooling, post-soldering phase of the process. It is also useful to assess mechanical strength of the parts and to reveal lots with excessive residual stresses.

A cold thermal shock in MLCCs caused by LN drop test resulted in a temperature decrease of capacitors that exceeded ΔT_c values determined using IWT for most of the part types. However, this test did not cause significant fracturing, and none of the tested samples had degraded leakage currents even after moisture exposure. This result can be explained by much worse heat transfer conditions in liquid nitrogen compared to cold water quenching. Measurements of the heat transfer coefficients by Lee at al. [19] showed that for the liquid nitrogen quench, the value of h is an order of magnitude lower than for the water quench. This is due to a rapid formation of a gaseous N2 film that immediately shields the surface of the sample immersed in liquid nitrogen, reduces transient stresses, and the probability of fracturing.

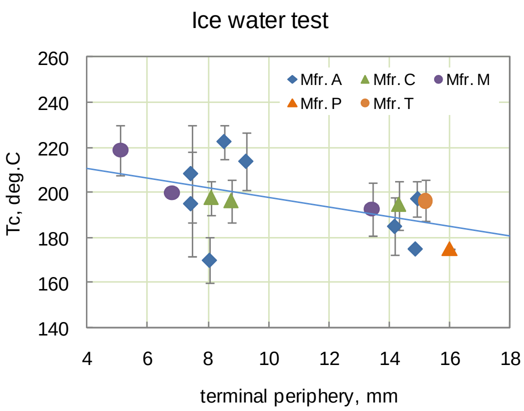


Figure 7. Effect of the length of terminal's periphery on critical temperature during IWT.

Conclusion

1. No degradation of electrical characteristics was observed after terminal solder dip testing up to 325°C, and only one sample out of forty had increased leakage currents after 350°C testing. A high robustness of MLCCs toward solder dip testing is mostly due to a high compressive strength of ceramic materials. Also, an increased thermal conductivity of capacitor along the metal plates mitigates the effect of solder dip stresses.

2. Ice water test is a reproducible and effective technique for evaluation of the susceptibility of MLCCs to fracturing during cold thermal shock conditions. Majority of the parts withstood IWT at 150 ^{o}C and the average critical temperature varied from 170 ^{o}C to 222 ^{o}C. The values of ΔT_c correspond to the tensile strength of the X7R capacitors in the range from 110 MPa to 200 MPa. Most of the TS-induced cracks formed at the terminal areas of the capacitors.

3. None of the geometrical factors of capacitors has a strong correlation with the critical temperature determined by IWT. However, there is a trend of decreasing of the thermal shock resistance with the terminal periphery of capacitors. This indicates that a combination of residual and TS-related stresses is the major cause of fracturing and failures of MLCCs.

4. In spite of significant temperature difference that capacitors experienced during cold thermal shock caused by the liquid nitrogen drop test, this testing resulted in much less severe damage in ceramic capacitors compared to the ice-water testing performed at conditions with similar temperature difference. This result is due to a rapid formation of a gaseous N2 film that shields the surface of the capacitors and significantly reduces the heat transfer from the part and confirms that the heat transfer conditions are of critical importance for TS testing and for the assessment of the thermo-mechanical reliability of ceramic capacitors.

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